DIVISION S-8—NUTRIENT MANAGEMENT & SOIL & PLANT ANALYSIS

Measuring Water-Extractable Phosphorus in Manure as an Indicator of Phosphorus in Runoff

Peter J. A. Kleinman,* Andrew N. Sharpley, Ann M. Wolf, Douglas B. Beegle, and Philip A. Moore, Jr.

ABSTRACT

Water-extractable P (WEP) in manure is correlated with P concentration in runoff from soils amended with manure and is, thus, an effective indicator of environmental P loss. This study sought to elucidate methodological factors affecting WEP measurement in manure and to quantify errors related to two established methods of manure WEP measurement. Dairy cow (*Bos taurus*) manure, poultry (*Gallus gallus* domesticus L.) (layer) manure, and swine (*Sus scrofa* domestica L.) slurry were used. Varying dry matter/distilled water ratios (1 to 20:200) revealed that greater dilution of manure dry matter increased WEP (mean 1.8–5.4 g kg^{-1}), likely because of the dissolution of calcium phosphates. Increasing shaking time from 1 min to 24 h, increased manure WEP concentration (average 3.7–8.2 g kg^{-1}). Filtration with Whatman 1 paper filters resulted in significantly higher WEP measurements in dairy and poultry manure (4.1 g kg^{-1}) than with a 0.45-μm filtration (3.7 g kg^{-1}). No significant difference was observed in the swine slurry. A rainfall-runoff experiment using simulated rainfall was conducted to determine the effect of the individual factors on predicting dissolved-reactive P (DRP) concentration in runoff. Comparison of regression coefficients relating manure WEP to runoff DRP concentration revealed an optimum shaking time between 30 min and 2 h, but did not support any single manure/distilled water ratio or filtration method. Replication of two established methods of manure WEP measurement resulted in coefficients of variation of 0.01 to 0.12. Results of this study support the use of a single method with a fixed manure/distilled water ratio for liquid and dry manures.

Widespread concern about freshwater eutrophication in the USA has led to the development of site assessment indices to aid in identifying critical source areas of P loss from agricultural watersheds (Lemunyon and Gilbert, 1993; Gburek and Sharpley, 1998). These indices differentiate between "source" and "transport" factors controlling P transfers from land to water, with source factors representing the pools or amount of P at a site and transport factors representing the potential to transport P from that site. Because recent additions of P in either mineral fertilizer or manure can greatly exacerbate runoff P losses (Kleinman, 2000; Sharpley et al., 1998), most states have adopted or are in various stages of developing site assessment indices that distinguish between mineral and manure sources of P on the basis of P availability to runoff water (Weld et al., 2000). These indices are a component of a national strategy to develop Comprehensive Nutrient Management Plans that considers P impacts and utilization for animal feeding operations (USDA and USEPA, 1999).

Soil and manure P solubility in water likely controls DRP concentrations in runoff (McDowell and Sharpley, 2001a). For instance, Pote et al. (1999) found that DRP concentrations in surface runoff were closely related to WEP concentrations in three acidic soils. Because manure application to soils results in large, temporary increases in WEP at the soil surface, the zone that serves as the source of P in runoff, forms of P added to soil directly affect P availability to runoff. Moore et al. (2000) reported significant differences in DRP concentrations in runoff from pastures amended with either alum-treated or untreated poultry litter. They observed concomitant decreases in the WEP fraction of poultry litter treated with alum and runoff DRP concentrations from the pasture receiving that litter. Others have also found a variation in DRP loss in runoff as a function of manure type (Sharpley et al., 1998; Westerman and Overcash, 1980). For instance, Kleinman et al. (2002) found the WEP concentration of dairy, poultry, and swine manure applied to the surface of three soils to be highly correlated with DRP losses in runoff.

At present, two methods of manure WEP determination have been reported in the literature: Self-Davis and Moore (2000) and, Sharpley and Moyer (2000). The Self-Davis and Moore method was developed for dry manure, particularly poultry litter, requiring a 20-g sample (wet weight) of manure to minimize error associated with obtaining a representative sample. This method extracts P from the sample by shaking for 2 h in 200 mL water. The Sharpley and Moyer method was originally used to determine WEP in manures with dry matter ranging from 11 to 89%, extracting 1 g (dry weight equivalent) of fresh manure with 200 mL water for 1 h (Sharpley and Moyer, 2000). Although both methods analyze fresh manure, the Self-Davis and Moore method

P.J.A. Kleinman and A.N. Sharpley, USDA-ARS, Pasture Systems and Watershed Management Research Unit, Curtin Road, University Park, PA 16802; A.M. Wolf, Agricultural Analytical Services Lab., The Pennsylvania State Univ., University Park, PA 16802; D.B. Beegle, Dep. of Crop and Soil Sciences, The Pennsylvania State Univ., University Park, PA 16802; P.A. Moore, Jr., USDA-ARS, Poultry Production and Product Safety Research Unit, Plant Sciences 115, Univ. of Arkansas, Fayetteville, AR 72701. Received 15 Jan. 2002. *Corresponding author (pjk9@psu.edu).

Published in Soil Sci. Soc. Am. J. 66:2009–2015 (2002).

Abbreviations: DRP, dissolved-reactive P; RCF, relative centrifugal force; TP, total P; WEP, water-extractable P.

calls for an extraction ratio of manure/distilled water on a manure wet weight measurement of manure, while the Sharpley and Moyer method employs a ratio based on dry-weight equivalency. As such, the methods vary in manure/distilled water ratio when applied to liquid manures.

While manure WEP determined by the above methods has been correlated with runoff DRP concentrations (Moore et al., 2000; Sharpley and Moyer, 2000; Kleinman et al., 2002), the two methods are sufficiently different to call into question the comparability of manure WEP results from these studies. Clearly, there is a need to critically evaluate these methods, in terms of their ability to estimate manure P solubility and potential P release to runoff water. Further, as manure management becomes an integral part of nutrient management planning that addresses water quality as well as crop production, there will be a need for more routine information on soluble manure P than on total P as is currently provided by state and private soil testing laboratories. Development of a simple accurate method for determining manure WEP that can be routinely conducted by analytical laboratories, will thus, greatly aid farm planners and advisors to more reliably manage land application of manures to minimize P runoff.

The general objective of this study was to evaluate select analytical parameters impacting the measurement of WEP in manure. Specifically, the roles of manure/distilled water, shaking time, and filtration method in determining WEP are assessed, as are their effects on the relationship between WEP and runoff DRP from soil recently broadcast with manure. Finally, two established methods of WEP determination are compared to assess their merits as manure testing procedures.

MATERIALS AND METHODS

Manure Sampling and Analysis

One dairy cow manure, one layer poultry manure, two broiler poultry manures (one treated with alum, one untreated), and one swine slurry were selected to represent a wide range of dry matter contents and expected P solubilities (Table 1). Dairy manure and swine slurry were sampled from the Pennsylvania State University Dairy and Swine Centers, respectively, at University Park, PA. The dairy manure was from lactating Friesian-style dairy cows that was scraped from a free stall barn. Swine slurry was from finishing sows that was washed into a holding tank and agitated before sampling. Manure from a poultry laying operation in Northumberland County, PA, was collected directly from the layer house. Poultry litter (wood shaving bedding) was collected from commercial broiler houses in northwest Arkansas that were either untreated or had received alum (1362 kg alum house^{-1}) (Moore et al., 2000). All manures were stored at 4°C in sealed plastic containers for 1 to 2 wk before analysis.

Table 1. Properties of manures used in study.

Manure type	Solids	Total N	Total P	pH
	%	g kg^{-1} (dry weight basis)		
Dairy manure	16	30	6	8.0
Layer poultry manure	53	35	23	8.9
Swine Slurry	2	117	33	7.3
Broiler poultry litter, untreated	76	41	14	8.1
Broiler poultry litter, alum treated	75	44	12	7.6

Manure was analyzed for total P (TP) by the modified semimicro-Kjeldahl procedure (Bremner, 1996). Manure pH was determined after mixing 1 g (equivalent dry weight) fresh manure with 100 mL of distilled water. Dry matter content of all manures was determined by gravimetric analysis (70°C basis).

Comparison of Established Manure Water-Extracted Phosphorus Protocols

Water-extractable P in the manure was determined by the method of Sharpley and Moyer (2000) and by a modified method of Self-Davis and Moore (2000). In the Sharpley and Moyer (2000) method, dry matter content of the manure was first determined gravimetrically. Then 1 g (dry-weight equivalent) of fresh manure was shaken end-over-end for 1 h in 200 mL of distilled water, followed by centrifugation (20 min at 2900 $\times$ g) and filtration through Whatman 1[1] filter paper (Whatman International Ltd., Maidstone, England). In the Self-Davis and Moore method, 20 g of manure (fresh weight) was shaken end-over-end in 200 mL of distilled water for 2 h. The supernatant was centrifuged at a relative centrifugal force (RCF) of 2900 $\times$ g for 20 min and filtered first through a Whatman 1 filter paper and then through a 0.45-μm syringe filter. Self-Davis and Moore employ a different method to measure WEP in liquid manures: centrifugation of the liquid manure, filtration, and acidification (P.A. Moore, Jr., personal communication, 2001). Because this method is not applicable to dry manures, it was not included in this study.

The original Self-Davis and Moore (2000) protocol includes acidification of the filtrate to pH 2 to prevent precipitation of calcium phosphates. As P determination was conducted immediately following filtration such that calcium phosphate precipitation between filtration and P determination was expected to be minimal, we did not acidify the filtrates. In fact, for each of the three manures, we compared acidified and unacidified subsamples from five filtrates and found no statistically significant difference ($p > 0.1$) in P concentration (data not shown).

For both the Sharpley and Moyer (2000) and Self-Davis and Moore (2000) methods, filtrate P was determined immediately after filtration by the colorimetric method of Murphy and Riley (1962). Laboratory error related to the replication of the Self-Davis and Moore (2000) and the Sharpley and Moyer (2000) methods was quantified for all manures. To ensure that batch error was well represented, and, hence the precision of each method adequately measured, each method was conducted, in duplicate, on 20 subsamples of each manure.

Effect of Methodological Variables on Manure Water-Extractable Phosphorus

To assess the effect of manure/distilled water ratio on WEP, duplicate samples of three manures in fresh condition (dairy, layer chicken, and swine slurry) were shaken end-over-end for 20 min at manure (grams, equivalent dry weight)/distilled water (mL) ratios of 20:200, 10:200, 5:200 and 1:200. As dry matter content of the three manures ranged widely, wet weights of the fresh manure varied within a single manure/distilled water ratio category, although the dry weight was held constant. Mixtures were centrifuged (20 min, RCF = 2900 $\times$ g), filtered through a Whatman 1 paper filter, and P

[1] Mention of trade names does not imply endorsement by the USDA.

determined. In addition, water-extractable Ca concentration in the supernatant was determined by inductively couple plasma-atomic emission spectroscopy (ICP-AES).

To assess the effect of shaking time on WEP, duplicate fresh samples of dairy manure, layer poultry manure, and swine slurry were extracted at a manure/distilled water ratio of 1:200 by mixing either 1 g (equivalent dry weight) in 200 mL of distilled water or 2 g (equivalent dry weight) in 400 mL of distilled water. The mixtures were shaken end-over-end and 15-mL subsamples were removed after 1, 5, 30, 60 120, 240, and 1440 min. The subsamples were centrifuged (RCF = 2900 × g for 20 min), filtered (Whatman 1), and filtrate P determined colorimetrically. Note that while manure/distilled water was constant between the two replicates, the volumes gradually decreased as subsequent samples were removed. To determine whether removal of subsamples over time significantly impacted WEP estimation, possibly by changing manure/distilled water with preferential sampling of either liquid or dry matter, we compared WEP estimated by either extracting 1 g (dry-weight equivalent) with 200 mL of distilled water, or 2 g (dry-weight equivalent) with 400 mL of distilled water. No significant difference was observed between the two methods ($p > 0.1$), justifying this experimental approach in evaluating the effect of time on WEP measurement.

To assess the effect of alternative methods of filtering the supernatant, 20 samples of the dairy, layer poultry, and swine manures were subjected to a single extraction at a manure/distilled water ratio of 1:200 with a shaking time of 1 h. Before P determination, subsamples were processed by (i) centrifuging (20 min, RCF = 2900 × g) and filtering through a Whatman 1 paper filter, and (ii) centrifuging (20 min, RCF = 2900 × g), filtering through a Whatman 1 paper filter and finally refiltering through a 0.45-μm filter.

Runoff Experiment

A runoff experiment was designed to evaluate the relationship between manure WEP concentration and runoff P losses using the National Phosphorus Research Project indoor runoff box protocol (National Phosphorus Research Project, 2001). The protocol employs stainless steel runoff boxes, 1-m long, 20-cm wide, and 5-cm deep with back walls 2.5 cm higher than the soil surface, and 5-mm drainage holes in the base (Kleinman et al., 2001). Cheese cloth is placed on the bottom of the box, followed by sufficient soil (Hagerstown silt loam) to achieve a bulk density of 1.3 to 1.5 g cm^{-3}. Runoff is generated by applying artificial rainfall on inclined (3%) soil runoff boxes using a TeeJet 1/2 HH SS 50 WSQ nozzle (Spraying Systems Co., Wheaton, IL) placed approximately 305 cm above the soil surface. Rainfall is delivered at approximately 7 cm h^{-1}, and has a coefficient of uniformity >0.83 within the 2 by 2 m area directly below the nozzle. Runoff is collected via a gutter, equipped with a canopy to exclude direct input of rainfall and inserted at the lowest edge of the runoff box.

The surface horizon (0–20 cm) of a Hagerstown soil (fine, mixed, semiactive, mesic Typic Hapludalf) was collected, field sieved (2 cm), air dried, and thoroughly mixed. The mixed soil was analyzed for Mehlich-3 P (Mehlich, 1984) by shaking 2.5 g of soil with 25 mL of Mehlich-3 solution (0.2 M CH_3COOH + 0.25 M NH_4NO_3 + 0.015 M NH_4F + 0.013 M HNO_3 + 0.001 M EDTA) for 5 min. The supernatant was filtered (0.45 μm) and P in the neutralized filtrate determined by the method of Murphy and Riley (1962).

Runoff boxes were packed with the Hagerstown soil and then amended (surface application) with either dairy manure, layer poultry manure, or swine slurry (Table 1) at a TP application rate of 100 kg ha^{-1}. All treatments were conducted in duplicate. In addition, two boxes with unamended soil served as controls. Within 72 h of the manure application, artificial rainfall was applied to the runoff boxes, the initial 30 min of runoff collected from each box and the volume determined. After thorough mixing and agitation of each sample, a subsample was immediately filtered (0.45 μm). Dissolved-reactive P was determined on the filtered sample by colorimetric P determination (Murphy and Riley, 1962) within 24 h of collection. Total P was measured on unfiltered runoff water by modified a semimicro-Kjeldahl procedure following Bremner (1996).

Statistical Analysis

Associations between manure/distilled water, extraction time, and manure WEP concentration were assessed by least squares regression as were corresponding associations between manure WEP and runoff DRP concentration (Neter et al., 1996). Differences related to filtration method were evaluated by Student's t-test. Descriptive statistics were used to assess error related to the replication of the Self-Davis and Moore (2000) method with the Sharpley and Moyer (2000) method (Snedecor and Cochran, 1991). All analyses were conducted using Minitab's statistical software, Release 11 (Minitab Inc., 1996).

RESULTS AND DISCUSSION

Individual Methodological Variable Effects on Water-Extractable Phosphorus

Manure/Distilled Water Ratio

The relationship between WEP and manure/distilled water was similar for all three manures. Namely, WEP concentration decreased as manure weight increased, indicating a positive effect of dilution by distilled water on manure WEP concentration (Fig. 1). As all three manures had pH values ranging from 7.3 to 8.9, it is likely that dilution of manure promoted the dissolution of insoluble calcium phosphates and, therefore, higher WEP concentrations. This hypothesis is supported by the similar relationship observed between manure/distilled water and water-extractable Ca concentration of

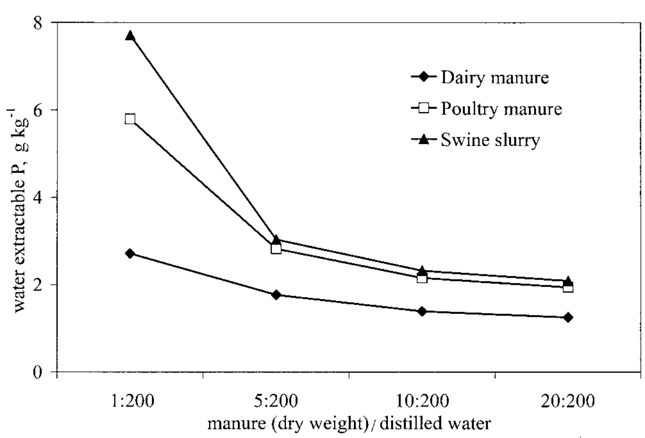

Fig. 1. Relationship of manure/distilled water to WEP concentration in dairy, poultry, and swine manures (mean of two observations).

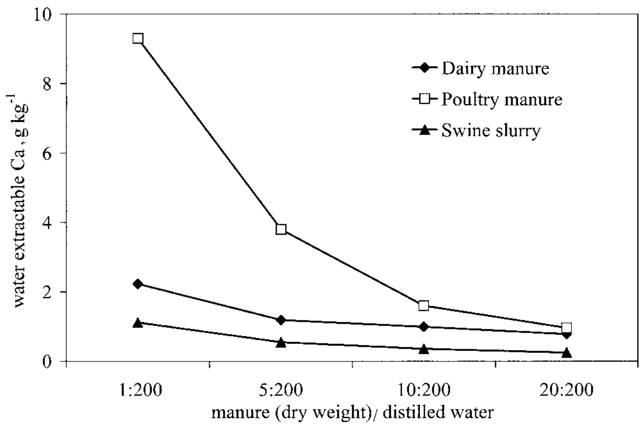

Fig. 2. Relationship of manure/distilled water to water extractable Ca concentration in dairy, poultry, and swine manures (mean of two observations).

the supernatant (Fig. 2). Greater concentrations of water-extractable Ca at lower manure/distilled water ratios are indicative of dissolution of Ca as well as P. In fact, WEP and water-extractable Ca concentrations were strongly associated within individual manure types (r^2 ranged from 0.97 to 0.99).

To assess the effect of manure/distilled water ratio on the prediction of runoff DRP from manure WEP, concentrations of DRP in runoff from the runoff experiment were related to manure WEP concentrations as determined at the various manure/water ratios examined in this study. In the manure-amended soils, DRP accounted for 51 to 73% of runoff TP concentration, whereas DRP accounted for only 13% of runoff TP concentration from the unamended control soil (Table 2). Clearly, soluble P from the manures served as the major source of runoff DRP from soils amended with manure. High TP concentrations in runoff from the unamended control (comparable with the TP concentrations in runoff from the dairy manure amended soil) are due to high rates of erosion from that soil (no surface cover) as well as to the high initial P concentration of the soil (Mehlich-2 P = 415 mg kg^{-1}). Runoff DRP concentrations varied widely across all treatments (from 0.53 to 13.94 mg L^{-1}). Best-fitting regression models relating manure WEP and runoff DRP were exponential for 5:200, 10:200, and 20:200 (r^2 = 0.97) and linear for 1:200 (r^2 = 0.95, Table 3). Thus, the nonlinear relationship between manure WEP concentration and manure/distilled water ratio (Fig. 1) affected the form of the regression equations but not the strength of the regressions (r^2).

Table 2. Results of rainfall-runoff experiment.

Treatment	Runoff DRP†	Runoff TP	Runoff volume
	—— mg L^{-1} ——		mL
Control (unamended soil)	0.53	4.22	4968
Dairy manure amended soil	2.26	3.54	3780
Poultry (layer) manure amended soil	10.97	21.26	5185
Swine slurry amended soil	13.94	18.97	5660

† Dissolved-reactive P.

Shaking Time

Manure WEP concentration is positively related to shaking time (Fig. 3), indicating that increasing the length of sample agitation in water releases more P to solution. This relationship is effectively described by a logarithmic model, suggesting that most WEP is extracted within the extraction period (24 h). In fact, >70% of WEP released by the three manures in 24 h was released in the first 60 min of extraction (Fig. 3).

Agitation serves to break down manure aggregates, exposing physically sequestered soluble P to solution and the longer shaking time allows for greater P desorption from mineral complexes. Regressions between manure WEP and runoff DRP concentration from the runoff experiment varied more widely with shaking time (r^2 ranged from 0.76 to 0.92) than with manure/distilled water ratio (r^2 ranged from 0.95 to 0.97) (Table 3). However, coefficients of determination (r^2) varied systematically with shaking time, increasing from 1 to 60 min and then decreasing above 60 min (Table 3). Based upon this experiment, an extraction time of 60 min provides the strongest regression coefficient and best predictor of runoff DRP.

Table 3. Effect of methodological factors on best fitting regression between manure WEP (g kg^{-1}) and runoff DRP (mg L^{-1}).

Extraction method	r^2	Regression equation
Manure/distilled water (shaking time = 20 min, paper filter)		
1:200	0.95	runoff DRP = 1.87WEP − 0.67
5:200	0.97	runoff DRP = $0.46e^{1.09WEP}$
10:200	0.97	runoff DRP = $0.46e^{1.42WEP}$
20:200	0.97	runoff DRP = $0.46e^{1.58WEP}$
Shaking time (manure/distilled water = 1:200, paper filter)		
1 min	0.76	runoff DRP = 2.51WEP + 0.01
5 min	0.88	runoff DRP = 2.41WEP − 1.11
30 min	0.90	runoff DRP = 1.79WEP − 0.77
60 min	0.92	runoff DRP = 1.46WEP − 0.50
120 min	0.91	runoff DRP = 1.43WEP − 0.70
240 min	0.87	runoff DRP = 1.38WEP − 0.73
1440 min	0.87	runoff DRP = 1.23WEP − 0.68
Filtration method (shaking time = 1 h; manure/distilled water = 1:200)		
Paper filter (Whatman 1)	0.96	runoff DRP = 1.93WEP − 0.47
0.45 μm filter	0.97	runoff DRP = 1.99WEP − 0.33
WEP Protocol		
Self-Davis and Moore (2000)	0.65	runof DRP = 1.29 WEP + 2.22
Sharpley and Moyer (2000)	0.97	runof DRP = 1.60 WEP + 0.17

Filtration Method

The effect of filtration method on WEP concentration varied with manure, apparently as a function of manure moisture content (Table 4). For the swine slurry (2% solids), there was no significant difference in WEP between extracts filtered through coarse paper filter (Whatman 1) and 0.45-μm membranes ($p = 0.82$). For dairy (16% solids) and poultry (53% solids) manures, WEP concentrations were significantly lower in 0.4-μm filtrates than paper filtrates ($p < 0.01$), accounting for 94 and 90% of WEP in the paper filtrate for dairy and poultry, respectively (Table 4). These differences point to the contribution of colloidal P to WEP in paper filtrates for these manures, which is removed by the 0.45-μm filter (Haygarth and Sharpley, 2000). The Murphy and Riley (1962) method may result in the hydrolysis of some P compounds associated with these colloids (McDowell and Sharpley, 2001b). Differences related to filtration may be even larger with ICP determination. Notably, regressions between manure WEP and runoff DRP varied little between the two filtration methods (Table 3), as relative differences in WEP concentration between the three manures remained sufficiently consistent to have no impact on runoff DRP prediction.

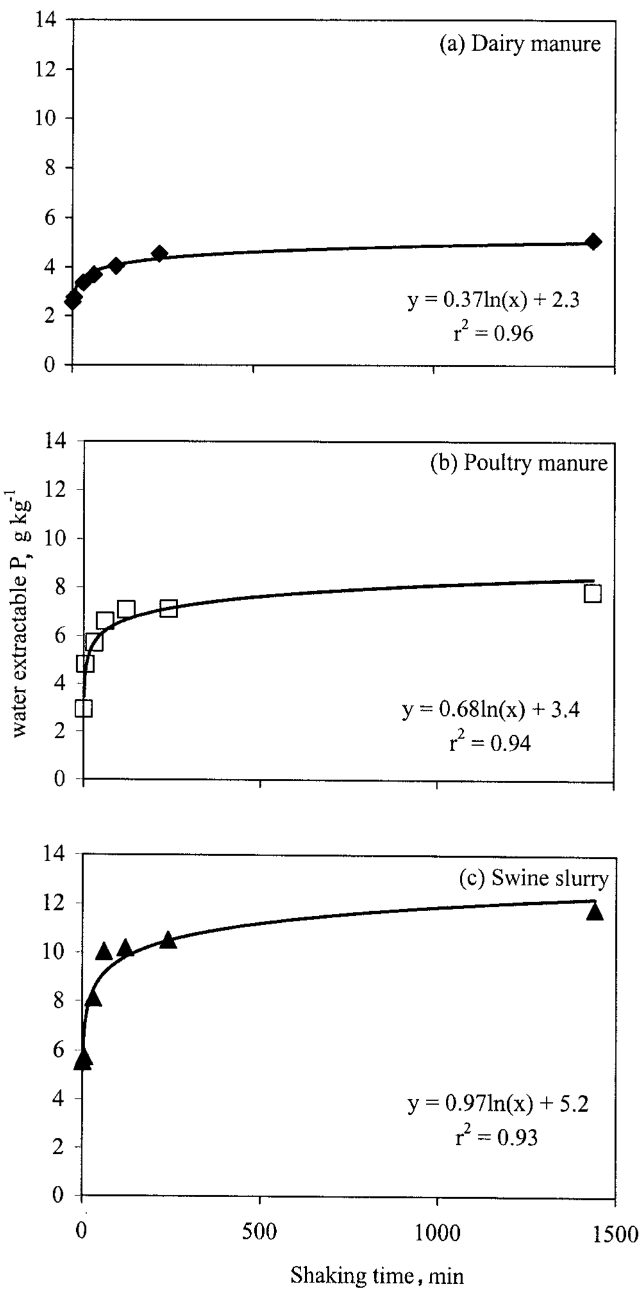

Fig. 3. Relationship between shaking time and WEP concentration for dairy manure, poultry manure, and swine slurry (mean of two observations).

Comparison of Established Water-Extracted Phosphorus Protocols

For most manures, coefficient of variations (CVs) were similar between the two methods (Table 5), ranging from 0.01 to 0.12 for the Self-Davis and Moore (2000) method and 0.06 to 0.12 for the Sharpley and Moyer (2000) method. These CVs are remarkably low, and comparable with CV obtained for routine soil P tests (Wolf and Baker, 1985; Sharpley et al., 1994; Kleinman et al., 2001).

Notably, WEP concentrations determined by the two methods vary considerably, with WEP determined by the Self-Davis and Moore (2000) method substantially greater than that by the Sharpley and Moyer (2000) method for the liquid manures, but considerably lower for the dry manures. These differences may be explained by the methodological variables examined earlier. The largest difference between the two methods is that the manure/distilled water ratio is held constant on a dry-weight equivalency basis by the Sharpley and Moyer (2000) method, and on a wet-weight basis by the Self-Davis and Moore (2000) method, which was developed for dry manures. While wet-weight determination involves less time and resources than predetermination of manure dry-matter content (and is therefore preferable for routine manure testing where efficient use of resources is a paramount consideration), the effect of using a wet-weight determination in the Self-Davis and Moore (2000) method is to vary the dry matter content of different manures and, thereby manure/distilled water ratio. Thus, for liquid manures, the manure/distilled water ratio was 0.4:200 for the swine slurry and 3:200 for the dairy manure. As described earlier, WEP concentration increases with greater dilution of manure in distilled water. Amongst the dry manures, manure/distilled water was 11:200 for the poultry (layer) manure and 15:200 for the two poultry (broiler) litters. Thus, for dry manures, the manure dry matter is more concen-

Table 4. Effect of filtration method on WEP measurement (shaking time = 1 h; manure/distilled water = 1:200).

Manure	Number of samples	Paper filter Mean	Paper filter s.d.†	0.45 μm filter Mean	0.45 μm filter s.d.
		g kg^{-1}			
Dairy	20	2.12	0.18	1.98	0.19
Poultry (layer)	20	6.05	0.37	5.43	0.26
Swine	20	7.14	0.43	7.15	0.53

† s.d. = standard deviation.

Table 5. Mean water-extractable P (WEP) concentration of manure and corresponding coefficient of variation for existing WEP methods.

	Self-Davids and Moore (2000)		Sharpley and Moyer (2000)		
Manure	Mean WEP (g kg^{-1})	Coefficient of variation	Mean WEP (g kg^{-1})	Coefficient of variation	Number of samples
Swine slurry	9.50	0.12	9.00	0.12	20
Dairy manure	2.56	0.11	1.92	0.10	20
Poultry (layer) manure	2.49	0.06	5.99	0.06	20
Poultry (broiler) litter, alum treated	0.20	0.01	1.51	0.10	20
Poultry (broiler) litter, untreated	0.59	0.07	3.91	0.10	20

trated in the Self-Davis and Moore (2000) method and WEP is accordingly lower than the Sharpley and Moyer (2000) method.

Other factors contributing to observed differences in the methods include extraction time and filtration method. The greater time of extraction called for in the Self-Davis and Moore (2 h) than the Sharpley and Moyer method (1 h) may account for the higher concentration of WEP in the dairy manure estimated by the former method, despite a comparatively greater concentration of dairy manure dry matter. In addition, the 0.45-μm filter used in the Self-Davis and Moore (2000) method may also contribute to the somewhat lower concentrations of WEP in the dry manures measured by that approach compared with Sharpley and Moyer (2000) (Table 5). In addition to filtering out P-containing colloids, the 0.45-μm filter may retain larger water-soluble compounds, such as labile condensed and polyphosphates, which are measured in the Murphy and Riley method and, more importantly, contribute to DRP transport in runoff.

The most important difference in the two methods is their ability to predict runoff DRP. The Self-Davis and Moore (2000) protocol results in comparatively poor regressions between manure WEP and runoff DRP concentration (Table 3), whereas the Sharpley and Moyer (2000) method produces very strong regressions. Undoubtedly, these differences are due to the varying manure/distilled water ratios associated with the Self-Davis and Moore (2000) protocol, and point to the imperative to maintain a fixed manure:distilled water ratio when comparing WEP concentration in different types of manures.

CONCLUSIONS

Results of this study support the development of a single method to measure WEP in manure. Existing methods of WEP measurement can be reliably replicated across a variety of manures. However, the ratio of manure dry matter to distilled water must be fixed to provide WEP measurements that are meaningful indicators of P availability to runoff water. Furthermore, best prediction of runoff DRP occurs with shaking times of at least 60 min. Filtration method, while affecting WEP measurement in drier manures, does not appear to significantly affect runoff DRP prediction.

Established WEP methods are well suited to the manures for which they were designed (e.g., Self-Davis and Moore, 2000, was originally developed for dry manures, such as poultry litter). However, given the wide range of manures that are analyzed by agricultural analytical laboratories, and based on the results of this study, the following rapid and reproducible method to estimate the potential of manure WEP to enrich runoff DRP is proposed.

Water-extractable Manure Phosphorus. Shake 1 g of dry-weight equivalent of fresh manure with 200 mL of distilled water on an end-over-end shaker for 60 min. Centrifuge mixture (about 2900 $\times$ g for 20 min to facilitate filtration) and filter. Determine P by the method of Murphy and Riley (1962). Water-extractable manure P is calculated as P concentration per unit dry-weight basis of manure (i.e., g WEP kg^{-1}).

REFERENCES

Bremner, J.M. 1996. Nitrogen—Total. p. 1085–1121. *In* D.L. Sparks (ed.) Methods of soil analysis. Part 3. SSSA Book Series no 5. SSSA and ASA, Madison, WI.

Gburek, W.J., and A.N. Sharpley. 1998. Hydrologic controls on phosphorus loss from upland agricultural watersheds. J. Environ. Qual. 27:267–277.

Haygarth, P.M., and A.N. Sharpley. 2000. Terminology for phosphorus transfer. J. Environ. Qual. 29:10–15.

Kleinman, P.J.A. 2000. Source risk indicators of nutrient loss from agricultural lands. p. 237–252. *In* M. Sailus (ed.) Managing nutrients and pathogens in animal agriculture. Northeast Regional Agricultural Engineering Service, Ithaca, NY.

Kleinman, P.J.A., A.N. Sharpley, K. Gartley, W.M. Jarrell, S. Kuo, R.G. Menon, R. Myers, K.R. Reddy, and E.O. Skogley. 2001. Interlaboratory comparison of soil phosphorus extraction methods. Commun. Soil Sci. Plant Anal. 32:2325–2345.

Kleinman, P.J.A., A.N. Sharpley, B.G. Moyer, and G.F. Elwinger. 2002. Effect of mineral and manure phosphorus sources on runoff phosphorus. J. Environ. Qual. (In press).

Lemunyon, J.L., and R.G. Gilbert. 1993. Concept and need for a phosphorus assessment tool. J. Prod. Agric. 6:483–486.

McDowell, R.W., and A.N. Sharpley. 2001a. Approximating phosphorus release from soils to surface runoff and subsurface drainage. J. Environ. Qual. 30:508–520.

McDowell, R.W., and A.N. Sharpley. 2001b. Soil phosphorus fractions in solution: Influence of fertiliser and manure, filtration and method of determination. Chemosphere 45:737–748.

Mehlich, A. 1984. Mehlich 3 soil test extractant: A modification of Mehlich 2 extractant. Commun. Soil Sci. Plant Anal. 15:1409–1416.

Minitab. 1996. Minitab user's guide, release 11. Minitab, State College, PA.

Moore, P.A., Jr., T.C. Daniel, and D.R. Edwards. 2000. Reducing phosphorus runoff and inhibiting ammonia loss from poultry manure with aluminum sulfate. J. Environ. Qual. 29:37–49.

Murphy, J., and J.P. Riley. 1962. A modified single solution method for the determination of phosphate in natural waters. Anal. Chim. Acta 27:31–36.

National Phosphorus Research Project. 2001. National research project for simulated rainfall—surface runoff studies. Available at http://www.soil.ncsu.edu/sera17/publications/National_P/National_P_Project.htm (verified 21 Sept. 2001). Southern Ext. Res. Activity Information Exchange Group 17. North Carolina State University, Raleigh, NC.

Neter, J.W., M.H. Kutner, C.J. Nachtsheim, and W. Wasserman. 1996.

Applied linear statistical models, 4th ed. Times Mirror Higher Education Group, Irwin, Chicago, IL.

Pote, D.H., T.C. Daniel, D.J. Nichols, A.N. Sharpley, P.A. Moore, Jr., D.M. Miller, and D.R. Edwards. 1999. Relationship between phosphorus levels in three ultisols and phosphorus concentrations in runoff. J. Environ. Qual. 28:170–175.

Self-Davis, M.L., and P.A. Moore, Jr. 2000. Determining water soluble phosphorus in animal manure. p. 74–76. *In* G.M. Pierzynski (ed.) Methods of phosphorus analysis for soils, sediments, residuals and waters. Southern Cooperative Series Bulletin #396, North Carolina State Univ., Raleigh, NC.

Sharpley, A.N., R. Indiati, C. Ciavatta, N. Rossi, and P. Sequi. 1994. Interlaboratory comparison of iron oxide-impregnated paper to estimate bioavailable phosphorus. J. Environ. Qual. 23:14–18.

Sharpley A.N., J.J. Meisinger, A. Breeuwsma, J.T. Sims, T.C. Daniel, and J.S. Schepers. 1998. Impacts of animal manure management on ground and surface water quality. p. 173–242. *In* J. Hatfield (ed.) Effective management of animal waste as a soil resource. Ann Arbor Press, Chelsea, MI.

Sharpley, A., and B. Moyer. 2000. Phosphorus forms in manure and compost and their release during simulated rainfall. J. Environ. Qual. 29:1462–1469.

Snedecor, G.W., and W.G. Cochran. 1991. Statistical methods, 8th ed. Iowa State University Press, Ames, IA.

USDA and USEPA. 1999. Animal Feeding Operations Unified Strategy. March 9, 1999. (Available online with updates at http://cfpub.epa.gov/npdes/afo/caforule.cfm?program_id=7) (verified 24 May 2002).

Weld, J.L., D.B. Beegle, J.T. Sims, J.L. Lemunyon, A.N. Sharpley, W.J. Gburek, F.C. Coale, and A.B. Leytem. 2000. Comparison of state approaches to the development of phosphorus indices. p. 329. *In* 2000 Agronomy abstracts. ASA, Madison, WI.

Westerman, P.W., and M.R. Overcash. 1980. Short-term attenuation of runoff pollution potential for land-applied swine and poultry manure. p. 289–292. *In* Livestock waste—A renewable resource. Proc. 4th Int. Symp. on Livestock Wastes. ASAE, St. Joseph, MI.

Wolf, A.M., and D.E. Baker. 1985. Comparisons of soil test phosphorus by Olsen, Bray P1, Mehlich-I, and Mehlich-3 soil test. Commun. Soil Sci. Plant Anal. 16:467–484.